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TITLE: MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE
DEAD CREEK SITE - QUALITY ASSURANCE

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ABSTRACT: This report summarizes the quality assurance results obtained for various samples analyzed during the course of this project. The accuracy (recovery from spiked samples) and precision (relative standard deviation of replicate determinations) results are tabulated herein. Although it would be difficult to summarize the overall performance of the methods for all the analytes, in general, the methods performed at the recovery and precision levels established during method validation.

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MEASUREMENT OF SELECTED CHEMICALS IN SOIL FROM THE DEAD CREEK SITE -
QUALITY ASSURANCEINTRODUCTION

Following media reports and subsequent Illinois EPA concern about hazardous chemicals at the Dead Creek site near Sauget, Illinois, personnel from Monsanto's W. G. Krummrich Plant sampled several areas at the site. Samples were submitted to Environmental Sciences for analyses for polychlorinated biphenyls, elemental phosphorus, chlorinated benzenes, chlorinated phenols, phosphate esters, and metals. During the various determinations, replicates and spiked samples were analyzed to evaluate the performance of the method used for these particular samples.

SUMMARY

This report summarizes the quality assurance results obtained for various samples analyzed during the course of this project. The accuracy (recovery from spiked samples) and precision (relative standard deviation of replicate determinations) results are tabulated herein. Although it would be difficult to summarize the overall performance of the methods for all the analytes, in general, the methods performed at the recovery and precision levels established during method validation.

DETAILSAnalytical Methods

The soil samples were analyzed for the various chemicals using established procedures or methods developed and validated for the chemicals of interest in soil. The following list tabulates the methods which were used.

Analyte	Method No.	Title
Polychlorinated Biphenyls	ES-80-M-28	Determination of Polychlorinated Biphenyls in Soil and Sediment
Chlorinated Benzenes	ES-80-M-29	Determination of Chlorinated Benzenes in Soil and Sediment
Chlorinated Phenols	ES-80-M-30	Determination of Chlorinated Phenols in Soil and Sediment
Elemental Phosphorus (P ₄)	ES-80-M-24	Determination of Elemental Phosphorus (P ₄) in Soil and Sediment
Phosphate Esters	ES-80-M-5	Determination of Group I Compounds in Sediments . . .
Metals	Ref. 1, 2	Inductively Coupled Plasma (ICP) . . . Method for Trace Element Analysis of Water and Wastes
Arsenic	Ref. 3	Methods for Chemical Analysis of Water and Wastes - Arsenic

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All determinations were carried out in strict accordance with these methods, except that the polychlorinated biphenyls, chlorinated benzenes and phosphate esters were measured in extracts from acidified samples to facilitate determination of chlorinated phenols in the same extracts.

Results

The results for the determinations of the compounds of interest have been reported in Special Studies ES-80-SS-24, 25, and 26, Measurement of Selected Chemicals in Soil from the Dead Creek Site . . . This Special Study is a compilation of the quality assurance results for all three Special Studies.

Quality Assurance

The recovery and precision results for the determinations are tabulated in Tables I-V. Each table contains the results for all quality assurance samples for a specific group of compounds. Recovery results are reported as percent recovery, calculated as

$$\% \text{ Recovery} = \frac{\text{Concentration (sample + spike)} - \text{average concentration (sample)}}{\text{Concentration (spike added)}} \times 100$$

Precision results are reported as percent relative standard deviation (RSD) for replicate determinations.

The tables present the recovery and precision results in concentration ranges (1-10 ppm to 10,000 - 100,000 ppm). The entries are averages of all values for all samples which had either recovery or precision evaluated in that range. All values are for actual samples except the metals recovery results, which are for spiked blank soil. In the recovery column, NE means Not Evaluated, i.e., no samples were spiked in that concentration range, and ND means Not Determinable, i.e., the spiking level was too low (usually <50%) compared to the level actually in the sample. In the precision columns, NE means Not Evaluated, i.e., no replicates were analyzed which contained the analyte in that concentration range.

More detailed compilations of the accuracy and precision results can be found in Reference 4.

REFERENCES

1. Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-79-020, page: Metals - 6, Section 4.1.3.
2. Federal Register, Vol. 44, No. 233, December 3, 1979.
3. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Method 206 - Arsenic, pages: 206.2-1 to 206.5-2.
4. RGK NBP 1914831

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TABLE 1. PCBs AND ELEMENTAL PHOSPHORUS (P₄) IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm		1,000-10,000 ppm		10,000-100,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
PCB's (Cl ₂ to Cl ₆ Homologs)		ND	NE	70%	17%	120%	18%	77%	58%	NE	0%
P ₄		56%	NE	NE	NE	NE	NE	NE	NE	NE	NE

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TABLE II. CHLOROBENZENES IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
MONOCHLOROBENZENE		105%	NE	110%	NE	100%	NE
P-DICHLOROBENZENE		120%	21%	125%	64%	120%	NE
O-DICHLOROBENZENE		125%	16%	120%	NE	120%	8%
TRICHLOROBENZENES (3)		96%	14%	110%	13%	120%	NE
TETRACHLOROBENZENES (3)		110%	9%	120%	NE	130%	NE
PENTACHLOROBENZENE		140%	12%	120%	NE	140%	NE
HEXACHLOROBENZENE		135%	13%	90%	NE	110%	NE
NITROCHLOROBENZENES (O-,P-)		125%	37%	120%	NE	120%	26%

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TABLE III. CHLOROPHENOLS IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
O-CHLOROPHENOL		19%	34%	64%	NE	58%	NE
P-CHLOROPHENOL		36%	26%	16%	NE	30%	NE
2,4-DICHLOROPHENOL		66%	47%	59%	NE	50%	NE
PENTACHLOROPHENOL		140%	46%	40%	NE	36%	20%

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TABLE IV. PHOSPHATE ESTERS IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		10-100 ppm		100-1,000 ppm		1,000-10,000	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
DIBUTYLPHENYL PHOSPHATE		75%	NE	130%	NE	120%	12%	NE	NE
BUTYLDIPHENYL PHOSPHATE		120%	42%	115%	NE	NE	NE	NE	NE
TRIPHENYL PHOSPHATE		120%	89%	120%	NE	115%	NE	NE	6%
2-ETHYLHEXYLDIPHENYL PHOSPHATE		90%	47%	110%	NE	115%	NE	NE	NE
ISODECYLDIPHENYL PHOSPHATE		NE	NE	NE	NE	NE	NE	NE	NE
T-BUTYLPHENYLDIPHENYL PHOSPHATE		70%	NE	92%	100%	100%	NE	NE	NE
DI-T-BUTYLPHENYLPHENYL PHOSPHATE		88%	NE	96%	NE	NE	NE	NE	NE
NONYLPHENYLDIPHENYL PHOSPHATE		84%	8%	76%	NE	96%	NE	NE	NE
CUMYLPHENYLDIPHENYL PHOSPHATE		62%	21%	76%	NE	88%	NE	NE	NE

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TABLE V. METALS IN DEAD CREEK SOIL SAMPLES

ANALYTE	LEVEL	1-10 ppm		1-100 ppm		100-1,000 ppm		1000-10,000 ppm		10,000-100,000 ppm	
		% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
SILVER		NE	NE	NE	46%	NE	NE	NE	NE	NE	NE
ALUMINUM		ND	NE	ND	NE	ND	NE	NE	6.5%	NE	NE
BARIUM		ND	NE	ND	NE	89%	37%	NE	0%	NE	NE
BERYLLIUM		98%	NE	98%	NE	94%	NE	NE	NE	NE	NE
BORON		0%	NE	65%	19%	81%	3%	NE	NE	NE	NE
CALCIUM		ND	NE	ND	NE	ND	NE	NE	8.3%	NE	7.5%
CADMIUM		89%	5.2%	97%	21%	96%	NE	NE	NE	NE	NE
COBALT		51%	NE	115%	6.5%	97%	5.1%	NE	NE	NE	NE
CHROMIUM		27%	NE	109%	20%	91%	6.4%	NE	NE	NE	NE
COPPER		0%	NE	143%	66%	90%	NE	NE	NE	NE	11%
IRON		ND	NE	ND	NE	ND	NE	NE	NE	NE	8.1%
MAGNESIUM		ND	NE	ND	NE	ND	NE	NE	7.8%	NE	NE
MANGANESE		ND	NE	ND	13%	ND	10%	NE	NE	NE	NE
MOLYBDENUM		53%	NE	83%	11%	83%	NE	NE	NE	NE	NE
SODIUM		ND	NE	ND	NE	ND	11%	NE	NE	NE	NE
NICKEL		0%	NE	108%	14%	91%	13%	NE	4.5%	NE	NE
LEAD		0%	NE	165%	21%	93%	NE	NE	6.5%	NE	NE
PHOSPHORUS		ND	NE	ND	NE	ND	10%	NE	17%	NE	7.9%
ANTIMONY		0%	NE	27%	2.9%	27%	13%	NE	NE	NE	NE
SILICON		ND	NE	ND	NE	0%	49%	NE	NE	NE	NE
TIN		88%	NE	85%	5.6%	96%	5.4%	NE	NE	NE	NE
STRONTIUM		81%	NE	105%	3.3%	94%	6.5%	NE	NE	NE	NE
TITANIUM		ND	NE	99%	30%	30%	1.3%	NE	NE	NE	NE
VANADIUM		ND	NE	ND	13%	120%	11%	NE	NE	NE	NE
ZINC		ND	NE	139%	34%	87%	NE	NE	8.9%	NE	16%
ARSENIC (By AA)		NE	NE	NE	NE	NE	NE	NE	NE	NE	NE

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